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# Mapping microplastic in Norwegian drinking water



Oregon State University



**Norsk Vann**

# About Norwegian Water

Norwegian Water is a special interest organisation representing Norway's water industry. It acts on behalf of the members, which are municipalities, companies owned by municipalities, municipal operational assistance organisations and some private water works. Norwegian Water in total represents 360 municipalities, with 95 % of the population. There are also nearly 100 affiliated members like consultants, producers, suppliers and institutions for research and education. Norwegian Water serves both as a special interest organisation and a competence building organisation for the members. The organisation works within the vision of "clean water – our future".

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# Norwegian Water Report

## Abstract

In the course of the project, 72 triplicate samples from 24 waterworks in Norway and 72 blanks were analysed for microplastic particles. From the findings in this study, it is concluded that concentrations of less than 4.1 microplastic particles per litre should not be given or used for comparison. Whenever analysis is done to elucidate a possible contamination of water, special care must be taken in the sampling and in the conductance of the analysis. Furthermore, the limits of detection and of quantitation must be taken into account in the design of the experiment, the sampling, and in the decision about the number and volume of samples to be analysed.

In the current study, no microplastic particles could be detected or quantified in the drinking water of the 24 water works who participated. They had been selected since their drinking water sources were anticipated to have the highest probability for all Norwegian water works to be polluted with microplastic particles.

Conclusively, it is very likely that microplastic particles cannot be detected in any drinking water in Norway. There is the small possibility that the drinking water in Norway contains microplastic particles at extremely low concentrations below the detection limit. However, these low concentrations do not provide a health risk.

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## Title

Mapping microplastic in Norwegian drinking water

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## Keywords

Microplastic, drinking water, sampling, analysis, public health, sources, occurrence.



# Preface



Norwegian Water and the authors like to thank all the water works involved, the Norwegian Ministry of Health and Care Services, the Norwegian Food Safety Authority and the Norwegian Institute of Public Health for participating in this project. Our joint efforts during this project have resulted in clear and distinct conclusions.

The study has been conducted by the Norwegian Institute for Water Research (NIVA). The health assessment was done by the Norwegian Institute of Public Health. Prof. Martin Jekel, Technische Universität Berlin, has conducted an evaluation and quality assurance check. His report and conclusions can be found in appendix 2.

The project has been funded by Norwegian Water and the participating water works.

A special thanks goes to Lars Hem, the City of Oslo, Agency for Water and Wastewater Services, for initiating this project together with Norwegian Water.

Hamar, 10.09.2018

Kjetil Furuberg  
Norwegian Water

# Summary in Norwegian

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## Sammendrag

### Bakgrunn og mål med prosjektet

Oppslag i ulike medier høsten 2017 satte fokus på forekomst av mikroplast i drikkevann globalt. Vannbransjen tar på alvor utfordringene med plastforsøpling og mikroplast, og tok derfor initiativ til å gjennomføre en norsk studie for å kartlegge forekomst av mikroplast i drikkevann. Prosjektet ble finansiert og ledet av Norsk Vann. Norsk institutt for vannforskning (NIVA) gjennomførte analyser og det vitenskapelige arbeidet. Folkehelseinstituttet bidro med vurdering av potensielle helseeffekter basert på resultatene. Prosjektet ble gjennomført i samarbeid med Mattilsynet og Miljødirektoratet. Rapporten fra prosjektet utgis på engelsk på grunn av stor interesse fra andre land knyttet til denne norske studien, som en av de første grundige kartleggingene av mikroplast i drikkevann.

### Deltagende vannverk

Totalt 24 vannverk deltok i studien, 20 med overflatevann og 4 med grunnvann som vannkilde. For alle vannverkene ble det tatt vannprøver både fra råvannet, fra behandlet vann og fra vann ute på ledningsnett. I studien ble det lagt vekt på å få kartlagt drikkevannet til en stor andel av befolkningen, og vannverkene ble også valgt ut fra en vurdering av risiko og representativitet. Følgelig er de vannverkene hvor det var forventet å finne de høyeste nivåene av mikroplast i vannkildene, også med i undersøkelsen.

### Prøvetaking og analyse av mikroplast

Mikroplast er plastbiter/fiber i størrelsesområde 0,1 mikrometer og opp til 5 millimeter. Det finnes ingen standard-metode for prøvetaking, kvantifisering og identifisering av mikroplast i ferskvann. Det er mange utfordringer knyttet til å bestemme konsentrasjonen av mikroplast i vann, blant annet fordi mikroplast er ujevnt fordelt i vannmassene, naturlig vann inneholder mange mikropartikler som ikke er mikroplast og omgivelsene inneholder mikroplast som kan forurense vannprøvene. Det ble gjennomført ulike tiltak for å unngå forurensning av prøvene under prøvetaking og analyse. Blant annet ble vannverkene bedt om å ta prøver på steder som er lite utsatt for støv og luftbevegelse og prøvetakeren måtte ikke ha på syntetiske klær. Prosedyrer ble fulgt for å la vannet renne før det ble tappet på godt skylte flasker. For hvert vannverk ble det tatt 3 parallelle 1 liters vannprøver både fra råvannet, fra behandlet vann og fra vann ute på ledningsnett. Hvert vannverk bidro derfor med 9 vannprøver som ble sendt til NIVA for analyse. Her ble vannprøvene analysert, i randomisert rekkefølge, ved først å filtrere vannet og deretter mikroskopere filtrerne for å visuelt bestemme antall mikroplastpartikler på filtrerne. Filtrerne var på forhånd undersøkt for mulig forurensning, og ulike prosedyrer ble fulgt for å unngå forurensning under analysen. For partikler < 60 mikrometer var det umulig å skille mikroplastpartikler fra andre partikler. Den benyttede metoden var derfor en analyse av mikroplast i størrelsesområdet 60-5000 mikrometer.

### Resultater og diskusjon

Totalt 72 blankprøver ble analysert innimellom analysen av de totalt 216 vannprøvene fra vannverkene. Blankprøvene bestod av filtrert, avionisert ultrarent vann (mikroplastfritt), tappet på samme type flasker som prøvene fra vannverkene. I gjennomsnitt 0,5 partikler/L (standardavvik 0,82/L) ble påvist i blankprøvene. Mikroplasten i blankprøvene stammet mest sannsynlig fra forurensning under selve analysen, i hovedsak fra luften i laboratoriet. Siden vannprøvene fra vannverkene ble utsatt for tilsvarende forurensning under analysen, ble gjennomsnittsverdier for blankprøvene trukket fra ved beregning av gjennomsnittsverdi for de ulike vannverksprøvene. Ut fra de 3 parallelle prøvene fra hvert prøvetakingssted fra de ulike vannverkene ble det beregnet gjennomsnittsverdier og et konfidensintervall (området hvor det med 67% sikkerhet kan sies at den reelle verdien av mikroplast i vannet ligger innenfor ved prøvetakingstidspunktet). Der hvor konfidensintervallet overlapper med null, kan man ikke si at mengde mikroplast i vannet hvor prøvene er tatt, er forskjellig fra null. For råvannsprøvene var det overlapp med null for prøvene fra 14 av 24 vannverk. For behandlet vann var det overlapp med null for hele 20 av 24 vannverk og for vannprøvene fra ledningsnett for 16 av 24 vannverk. Også for prøvene der 67% konfidensintervall ikke overlappet med null, ble det påvist svært lave mengder mikroplastpartikler, med gjennomsnittsverdier godt under 4,1 partikler/L som ble beregnet som nedre grense for å kunne kvantifisere antallet. Kun ett prøvepunkt fra ledningsnett hadde høyere gjennomsnittsverdi (5,5 partikler/L), men dette prøvepunktet viste seg å være lokalisert på et sted der forurensning fra luft var svært sannsynlig.

Det store antallet blankprøver og vannprøver (i triplikat) som ble analysert i studien gjorde det mulig for forfatterne å definere begrensningene for en slik vannanalyse. Resultatene la grunnlag for å bestemme nedre grense for deteksjon (0,9 partikler/L) og nedre grense for å kunne kvantifisere antallet (4,1 partikler/L) når tre parallelle 1 liters vannprøver analyseres som i denne studien, med samme metodikk og samme tiltak for å unngå forurensning. På grunn av metodens usikkerhet må gjennomsnittsverdier < 0,9 partikler/L oppgis som mikroplast ikke påvist. Gjennomsnittsverdier over deteksjonsgrensen, men med < 4,1 partikler/L bør ikke oppgis med antall og dermed ikke benyttes for sammenligning av prøver.

### Vurdering av helserisiko

Kartleggingsstudien viste at mengden mikroplast i drikkevann, både råvann, behandlet vann og vann på ledningsnettet, er nær null eller null. Det er behov for å utvikle standardiserte metoder for analyse av plastpartikler < 60 mikrometer for å bekrefte lave nivåer også av disse mindre plastpartiklene. Mennesker eksponeres for nano- og mikroplast gjennom mat og luft. Spesielt fisk og sjøvann er vist å inneholde betydelige mengder mikroplast. På det nåværende tidspunkt er det ikke tilstrekkelig med data om forekomst, toksisitet og opptak for å gjennomføre en full risikovurdering av om eksponering for nano- og mikroplast utgjør en fare for mennesker. Kartleggingsstudien viste at mikroplast fra drikkevann vil bidra svært lite til den samlede mengden av mikroplast som mennesker utsettes for. Med utgangspunkt i foreliggende kunnskap, vurderer Folkehelseinstituttet at disse lave nivåene av mikroplast i drikkevann utgjør ingen helsemessig risiko. Kampen mot plastforsøpling og mikroplast er likevel viktig for å hindre forurensning av miljøet.

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# 1. Background and objective

The presence of microplastic particles in aquatic ecosystems has been a topic broadly discussed by environmental organizations and authorities for some time. Reports on microplastic particles in drinking water have attracted attention recently.

The Guardian published a report on 06.09.2017 that claims the presence of microplastics in drinking water. This cross-border research tested 159 drinking water samples from five continents and found out that 83% of them were contaminated with tiny plastic debris. The findings were distributed and discussed by the international and the Norwegian press while scientists working on the analysis and treatment of drinking water jointly doubted whether the methods that had been applied were adequate to report such low concentrations as was done. The conclusion was that more investigations need to be performed to understand the extent of the problem and to make an adequate risk assessment. The Norwegian minister of Climate and Environment, Vidar Helgesen, clearly stated in Dagbladet, on 07.09.17, that a survey of the presence of microplastics in drinking water must be initiated immediately. A dialogue regarding this issue was initiated with The Ministry of Health and Care Services. The Norwegian Food Safety Authority was then commissioned to carry out this survey in cooperation with the Norwegian Institute of Public Health. Parallel to this, the City of Oslo, Agency for Water and Wastewater Services and Norwegian Water initiated a project to map the amount of microplastics in drinking water. Several Norwegian waterworks reported their interest in participating in such a survey. After a short dialogue, the two initiatives were merged and the project was funded by Norwegian Water and the participating waterworks.

There are currently no comprehensive/systematic studies available on microplastics in raw water resources, their behaviour during drinking water treatment, and their potential occurrence in drinking water. Therefore, the objective of the project was to determine the concentration of microplastics in drinking water by sampling and analysis of raw water, treated water, and tap water from a variety of waterworks in Norway (24 waterworks in total). The implementation of such a project should help to understand whether Norwegian drinking water contains microplastic particles and, if so, which health risk results from them and which measures should be taken to remove them, in order to guarantee the delivery of healthy drinking water to the consumers at every time.



## 2. Microplastics sources and occurrence

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### 2.1. Microplastics as contaminants of emerging concern

The term 'microplastics' commonly refers to plastic particles with upper size limit of 5 mm and without specified lower limit. The upper limit of 5 mm is generally accepted because this size is able to include a range of small particles that can be readily ingested by organisms. However, it has been suggested that the term microplastics be redefined as items <1 mm to include only particles in the micrometre size range, and the term 'mesoplastic' introduced to account for items between 1 and 5 mm. The following updated definition by Koehler et al. (2015) was reported accordingly:

- Macroplastics as >5 mm
- Mesoplastics as <5 mm to >1 mm
- Microplastics as <1 mm to >0.1 µm
- Nanoplastics as <0.1 µm

It is also common to distinguish between primary and secondary microplastics. Primary microplastics are manufactured as such and are used either as resin pellets to produce larger items or directly in cosmetic products such as facial scrubs and toothpastes or in abrasive blasting (e.g. to remove lacquers). Secondary microplastics are derived from fragmentation or disintegration of large plastic debris.

Microplastics have been present in the environment for many years. For instance, Carpenter et al. (1972), Colton, Burns, and Knapp (1974), and Gregory (1977) reported on marine plastics in the 1970s, but they have not been extensively studied, particularly in the context of freshwater systems. As research has focused on the issue more intensively since the early 2000s, microplastics are considered as contaminants of emerging concern.

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### 2.2. Sources of microplastics into the freshwater environment

Plastics will enter freshwater environments from various sources through various routes. On land, littering is an important environmental and public issue and is a matter of increasing concern in protected areas where volumes are influenced by visitor density. In addition, waste management practices in different regions of the world also vary, and this may be a more important source in one geographical region compared to another. As with bulk plastic items, microplastics can enter the environment by a number of pathways, and an important route in one geographical region may be less important in another.

Among all, passage through wastewater treatment plants (WWTPs) is considered one of the important sources of microplastics into freshwater systems. Examples of such particles include primary microplastics, used in personal care products, or fibres released from textiles during the washing of clothes. The overall retention capacity of WWTP was reported between 90-99% for particles of 20 to 300 µm diameter and higher efficiency for larger particles (Storck, Kools, and Rinck-Pfeiffer 2015, Talvitie et al. 2017). However, despite the high removal performance, even an advanced WWTP may constitute a considerable source of microplastics into the aquatic environment given the constant large volumes of effluent being discharged. Other sources which may contribute to the release of large volumes of synthetic particles include application of biosolids from WWTPs to agricultural lands, storm water overflow events, release from industrial products or processes, atmospheric deposition of fibres, emissions from construction sites, and tyre wear particles (Wagner and Lambert 2018). Once microplastics are released into the freshwater systems, most of them will be transported to oceans by rivers while the remaining particles will either be deposited in sediments and filtered in sand banks or reside in isolated water systems like remote mountain lakes.

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## 2.3. Occurrence in freshwater systems

Accumulation of microplastics in the marine environment has been well documented in the literature, whereas freshwater and estuarine systems have received less attention. Recent monitoring studies have established that, similar to marine environments, microplastics are found in a variety of freshwater matrices all over the world. Sampling of the Rhine River, Germany using a Manta net (Mani et al. 2015), microplastics were found in concentrations of about 900 thousand particles per km<sup>2</sup> with a peak concentration of 3.9 million particles per km<sup>2</sup>. Similarly, high microplastics concentrations were reported at the Three Gorges Dam, China (about 200 thousand to 14 million particles per km<sup>2</sup>), which were attributed to a lack of wastewater treatment facilities in smaller towns, as well as infrastructure issues when dealing with recycling and waste disposal (Zhang et al. 2015). However, the validity of these investigations must be doubted, as concentrations in water should be determined per volume and it is not possible to derive from the two publications, how the surface area of water had been sampled or how the conversion had been done.

Table 1 provides a brief overview of the environmental occurrence of microplastics in freshwater matrices in Asia, Europe, America, and Africa. However, it should be mentioned that the isolation of microplastics in environmental matrices can be highly challenging and detection and analytical confirmation of microplastics requires access to sophisticated equipment such as  $\mu$ -Fourier-transform infrared (FTIR) and  $\mu$ -Raman (see Chapter 3 for further details), which was not available in many of the studies performed. Therefore, many of these studies may underestimate the actual microplastic concentrations because their separation and identification are based on visual observation methods and may exclude those in the submicron size ranges.

Moreover, a comparison of data from different regions can be challenging due to the difference in sampling methods used, size ranges investigated, and the reporting units that are employed. Therefore, it is urgently needed to adopt universal criteria for sampling and reporting occurrence data of microplastics to facilitate a comparison. Additionally, the abundance of microplastics from different regions differs by several orders of magnitude. Even within the same region, the abundance of microplastics varies considerably. This uneven distribution pattern can be related to their relatively low density, which means that they can be transported easily by currents and accumulate in areas with weaker hydrodynamic condition (Wagner and Lambert 2018). In addition, the loading rate of plastic waste can differ significantly in different regions. Previously, Yonkos et al. (2014) demonstrated that the abundance of microplastics was positively correlated with population density and proportion of urban/suburban development within the watersheds. However, researches also demonstrated that microplastics were also found at relatively high concentrations in inland waters from remote areas with limited human activities (Free et al. 2014, Zhang et al. 2016). This is likely due to a lack of proper waste management measures in those areas.

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## 2.4. Occurrence in drinking water

Water suppliers using surface water as a raw water resource are likely to be affected by the potential presence of microplastic particles. Larger particles, as investigated in many studies, will presumably be retained during depth filtration, bank filtration, artificial recharge, or underground passage and membrane filtration (Storck, Kools, and Rinck-Pfeiffer 2015). Data on the occurrence of very small microplastic particles in freshwater systems and their behaviour during water treatment are still lacking at this stage. There are currently no comprehensive studies on microplastics in raw water resources, their behaviour during drinking water treatment, and their potential occurrence in drinking waters. The Helmholtz Centre for Polar and Marine Research (Alfred-Wegener-Institute, Bremerhaven, Germany) investigated the possible occurrence of microplastics in tap water in the supply area of the OOWV (Oldenburgisch- Ostfriesischer Wasserverband) (Mintenig, Löder, and Gerdt 2014). Five waterworks were included in this study, in which all had ground water as a source of drinking water. Samples were taken from untreated groundwater, processed drinking water, as well as two samples in one representative household per supply area.

Table 1. Studies on microplastics contamination in natural freshwater systems.

Location	Size (mm)	Abundance	Main types	Reference
Asia				
Three Gorges Dam, China	0.048 – 5.0	Mean: 4073 p/m <sup>3</sup>	PS, PP,PE	Di and Wang (2018)
		Max: 12611 p/m <sup>3</sup>		
Three Gorges Dam, China	0.112 - 5.0	Mean: 8465.6 × 10 <sup>3</sup> p/km2	PE, PP, PS	Zhang et al. (2015)
		Max: 13.617.5 × 10 <sup>6</sup> p/km2		
Taihu lake, Plankton net samples, China	0.333 – 5.0	Mean: -	CP, PA, PET, PP	Su et al. (2016)
		Max: 6.8 × 10 <sup>6</sup> p/km2		
Taihu lake, surface water, China	0.005 – 5.0	Mean: -	CP, PES, PA, PET, PP	Su et al. (2016)
		Max: 25.8 p/L		
Urban waters of Whuan, China	0.05 – 5.0	Mean: -	PA, PE, PET, PP, PS	Wang et al. (2017)
		Max: 8.925 × 10 <sup>3</sup> p/m <sup>3</sup>		
Yangtze river estuary, China	> 0.5	Mean: 4137.3 p/m <sup>3</sup>	Not identified	Zhao et al. (2014)
		Max: 1.02 × 10 <sup>4</sup> p/m <sup>3</sup>		
Coastal water, East China sea	0.5-5.0	Mean: 0.167 p/m <sup>3</sup>	Not identified	Zhao et al. (2014)
		Max: -		
Lake Hovsgol, Mon-golia	> 0.333	Mean: 20,264 p/km2	Not identified	Free et al. (2014)
		Max: 4.4435 × 10 <sup>4</sup> p/km2		
Three urban estuar-ies, China	> 0.5	Mean: -	PE, PP, PVC, PTFE	Zhao, Zhu, and Li (2015)
		Max: 4100 p/m <sup>3</sup>		
Europe				
Dutch river delta and Amsterdam canals	0.01-5.0	Mean: 100 p/L	Not identified	Leslie et al. (2017)
		Max: 187 p/L		
Rhine river, Germany	> 0.3	Mean: 892 777 p/km <sup>2</sup>	PS, PP, PS, PVC	Mani et al. (2015)
		Max: 3.9 × 106 p/km <sup>2</sup>		
Seine and Marne rivers, France	> 0.08	Mean: 30 p/m <sup>3</sup>	Not identified	Dris et al. (2015)
		Max: 106 p/m <sup>3</sup>		
Lake Geneva, Switzerland	0.3-5.0	Mean: -	PS (no detailed analysis)	Faure et al. (2012)
		Max: 48,146 p/km2		
Bolsena and Chiusi lakes, Italy	0.3-5.0	Mean: -	Not identified	Fischer et al. (2016)
		Max: 4.08 p/m <sup>3</sup>		
Danube river, Austria	0.5-2.0	Mean: 316.8 p/1000 m <sup>3</sup>	Not identified	Lechner et al. (2014)
		Max: 141647.7 p/1000 m <sup>3</sup>		
Tamar estuary, England	0.3-5.0	Mean: 0.028 p/m <sup>3</sup>	PE,PP, PS, PVC	Sadri and Thompson (2014)
		Max: -		
America				
Four estuaries in the Chesapeake Bay, USA	0.3-5.0	Mean: -	PE	Yonkos et al. (2014)
		Max: 259,803 p/km <sup>2</sup>		
North shore channel, Chicago, USA	> 0.333	Mean: 730,341 to 6,698,264 p/km <sup>2</sup> (range for upstream and down stream)	Not identified	McCormick et al. (2014)
		Max: -		

Lake Winnipeg, Canada	> 0.333	Mean: -	Not identified	Anderson et al. (2017)
		Max: $7.48027 \times 10^5$ p/km <sup>2</sup>		
29 great lakes tributaries, USA	> 0.333	Mean: 4.2 p/m <sup>3</sup>	Not identified	Baldwin, Corsi, and Mason (2016)
		Max: 32 p/m <sup>3</sup>		
Goiana stuary, Brazil	> 0.3	Mean: 0.031 to 0.26 p/100 m3	Not identified	Lima, Costa, and Barletta (2014)
		Max: 15.3 P/100m <sup>3</sup>		
Laurentian great lakes, USA	0.333-5.0	Mean: 43,157 p/km <sup>2</sup>	PE (No detailed analysis)	Eriksen et al. (2013)
		Max: $4.663 \times 10^5$ p/km <sup>2</sup>		
Los Angeles river, San Gabriel river, Coyote creek, USA	> 0.333	Mean: Vary greatly	Not identified	Moore, Lattin, and Zellers (2011)
		Max: $1.2932 \times 10^4$ p/m <sup>3</sup>		
Africa				
Five urban estuaries of KwaZulu-Natal, South Africa	> 0.250	Mean: 11.0 – 70.3 p/10,000 L	PS	Naidoo, Glassom, and Smit (2015)
		Max: 487 p/10,000 L		

*Abbreviations: PVC: polyvinylchloride; PE: polyethylene, PS: Polystyrene, PP: Polypropylene, PA: Polyamide, PET: Polyethylene Terephthalate, CP: Cellulose Propionate, PES: Polyether Sulfone, PTFE: Polytetrafluoroethylene, p: particles*

Microplastics in the form of fibres were detected in blank samples, implying a contamination through the exposure of the water samples through laboratory air. These data were used to correct the amount of fibres for each sample and it was considered that none of the fibres were originally in ground and drinking water samples. Only 24 particles made of polyvinylchloride (PVC), polyethylene (PE), polyamide (PA), polyester (PEST), and epoxy resin with sizes between 50 and 150 µm were found in 10 from the overall 24 samples. The calculated concentrations were very low and ranged from 0.4 to 7 microplastic particles per m<sup>3</sup> (0.0004 to 0.007 particles per litre) in ground or tap water for the ten samples. The authors affiliated these microplastics to the abrasion of pipes and fittings used in the drinking water system.

In addition, two recent studies reported the presence of microplastics in drinking water. Orb Media, a non-profit journalism organization, published a report that claims the presence of microplastics in tap water (termed as drinking water) from a number of countries around the world, such as Uganda, Indonesia, Lebanon, the United States, and Ecuador, as well as Europe (Kosuth et al. 2017). From Europe, there are samples from the UK, Germany, and France. The results showed that 83% of a total of 159 samples contained microplastics. The most frequent occurrence of microplastics was found in the United States and Lebanon. The majority of the microplastic particles found were fibres (99.7%) with the colours blue, black, red, brown, and transparent and lengths in the range of 100-5000 µm. The number of particles found was between 0 and 57 particles per litre with an average of 4.34 particles per litre.

Another study of microplastic in drinking water has been carried out by CPHBusiness Laboratory and Environment (CPHBusiness (2017)). The survey has been conducted as a pilot study with screening of drinking water taken as tap water from 16 investigated households in the Copenhagen area. The results showed that all samples contained microplastics with an average of 18 pieces of microplastics per litre of water (range 2 to 45).

## 2.5. Occurrence in bottled mineral water and other beverages

Few studies about synthetic particles in German beers were performed. The authors found fibres in the blank and the beverage samples, but the results did not show statistically significant differences, and some explained the identification of microplastics in German beer as an artifact of laboratory contamination. Recently, Schymanski et al. (2018) tested the microplastic content of water from 22 different returnable and single-use plastic bottles, 3 beverage cartons, and 9 glass bottles obtained from grocery stores in Germany. Small (50-500 µm) and very small (1-50µm) microplastic fragments were found in every type of water. Almost 80% of all microplastic particles found had a particle size between 5-20 µm (not detectable by many previous methods). A statistically significant difference from the blank value to the investigated packaging types could only be shown comparing to the returnable bottles. Table 2 summarizes the findings from these studies.

Table 2. Studies on microplastics contamination in mineral water and other beverages.

Sample	Size (µm)	Abundance in sample	Abundance in blank	Main types	Reference
Beer/Pilsener - bottle Beer/Wheat - bottle Beer/Alcohol free - bottle	-	25±21 (n=16) 26±25 (n=5) 17±13 (n=9)	No blank	Not ident	Liebezeit and Liebezeit (2014)
Beer - bottle	-	16±15 fibres (n=39) 21±16 fragments (n=39) 27±10 granules (n=39)	15±9 (n=10) 20±13 (n=10) 19±12 (n=10)	Not ident	Lachenmeier et al. (2015)
Beer/Pilsener - bottle	1-5000	30-57 (n=3)	30-57 (n=?)	PE, PS	Wiesheu et al. (2016)
Mineral water - Returnable Mineral water - Single use Mineral water - cartons Mineral water - glass bottles	1-500	118±88 (n=12) 14±14 (n=10) 11±8 (n=3) 50±52 (n=9)	14±13 (n=18)	PET, PP PET, PE PE, PET, PP PET, PE, PA	Schymanski et al. (2018)

Abbreviations: PE: polyethylene, PS: Polystyrene, PP: Polypropylene, PET: Polyethylene Terephthalate, PA: Polyamide.

A very recent study was performed by the State University of New York at Fredonia with a focus on microplastics contamination in bottled drinking water (Mason, Welch, and Neratko (2018)). In total 259 bottles of water from 11 brands were processed across 27 different lots (an identification number assigned by a manufacturer to a particular production unit) purchased from 19 locations in 9 countries. All samples were packaged in plastic except for one which was packaged in glass. All bottles had plastic bottle caps. An average of 10.4 microplastic particles >100µm per litre of bottled water was detected and confirmed by Fourier transform infrared (FTIR) analysis. Including smaller particles (6.5-100 µm), an average of 325 microplastic particles per litre was detected. These small particles were detected by using Nile red tagging alone and no spectroscopic confirmation was performed. The most common polymer among particles >100 µm was polypropylene which matches a common plastic used for the bottle cap. The authors suggested that contamination is at least partially coming from the packaging and/or the bottling process itself.



## 3. Analysis of microplastics

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### 3.1. General remarks

The analysis of microplastics is a new challenge for analytical scientists. The small size of microplastics complicates their determination in environmental samples compared to macroplastics and demands more sophisticated analytical approaches. Microplastics are heterogeneously distributed in the environment, and therefore representative sampling methods are essential. The sample matrix, independent of the sampled environmental compartment, contains a high burden of particles of natural origin that strongly interfere with the visual detection of microplastics. Therefore, suitable methods for the sample preparation are needed to extract microplastics and reduce the number of natural particles. Moreover, an analytical method for the identification and confirmation of the plastic particles is mandatory to obtain reliable results. However, no standardized method has been established so far for effective sampling, quantification, and identification of microplastics in freshwater samples (Li, Liu, and Chen 2017). Table 3 presents a summary of methodologies used by different research teams to analyse microplastics in freshwater systems.

### 3.2. Sampling methods

Sampling methods similar to those used in marine systems, are used to collect microplastics in freshwater systems (Eerkes-Medrano, Thompson, and Aldridge 2015). They include selective sampling and bulk or volume reduced sampling. Selective sampling has been applied to sediments, while bulk or volume-reduced sampling has been used in sampling both sediments or water parcels. **Bulk samples** refer to samples where the entire volume of the sample is taken without reducing it during the sampling process. Only a few studies collected bulk water samples from freshwater systems for analysis.

**Volume-reduced samples** refers to samples where the volume of the bulk sample is usually reduced during sampling, preserving only that portion of the sample that is of interest for further processing. This method is applied the most for sampling from lakes and rivers where microplastic particles were often sampled with Neuston or plankton nets or Manta trawls. Manta trawls with mesh sizes of 333 µm are used the most. This approach leads to nonquantitative sampling of microplastics with particle sizes of <300 µm. The nets with smaller mesh sizes are prone to clogging. To overcome this problem, some methods are being developed using filter cascades that result in a size fractionation during the sampling and the reduction of the matrix burden of the small mesh sizes (Löder and Gerdtz 2015). However, application of mesh sizes of 300 µm leads to nonquantitative sampling of microplastics with particle sizes <300 µm which should not be ignored.

### 3.3. Purification and removal of natural debris

The identification of microplastic particles is often prevented by natural debris that is present in the sample and accompanies the microplastics during the sampling of water samples. Thus, the destruction of natural debris or biological material is unavoidable to minimize the possibility of misidentification or underestimation of small plastic particles. The destruction of natural material can be carried out by chemical or enzymatically catalysed reactions.

Chemical destruction of natural debris is achieved through the treatment of the sample with hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), mixtures of hydrogen peroxide and sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), and Fenton-like reactions (Wagner and Lambert 2018). These harsh conditions might result in losses of plastics that are labile to oxidation or unstable in strong acidic solutions, such as poly (methyl methacrylate) or polycarbonates. To avoid the loss of synthetic polymers, which are not resistant against acidic treatments, usage of sodium hydroxide (NaOH) was proposed.

Table 3. Summary of methodologies for analysis of microplastics in freshwater systems.

Location	Collection	Mesh (µm)	Depth	Digestion	Identification	Reference
Asia						
Three Gorges Dam, China	Teflon pump and sieve	48	1 m	30% H <sub>2</sub> O <sub>2</sub>	Visualization + µ-Raman	Di and Wang (2018)
Three Gorges Dam, China	Trawl	112	-	-	FTIR	Zhang et al. (2015)
Taihu lake, China	Nylon Plankton net	333	30 cm	30% H <sub>2</sub> O <sub>2</sub>	µ-FTIR + SEM-EDS	Su et al. (2016)
Taihu lake, China	Bulk water 5 L	-	-	30% H <sub>2</sub> O <sub>2</sub>	µ-FTIR + SEM-EDS	Su et al. (2016)
Urban waters of Whuan, China	Teflon pump and steel sieve	50	0-20 cm	30% H <sub>2</sub> O <sub>2</sub>	FTIR	Wang et al. (2017)
Yangtze river estuary, China	Teflon pump and steel sieve	32	1 m	30% H <sub>2</sub> O <sub>2</sub>	Visualization	Zhao et al. (2014)
Coastal water, East China sea	Neuston net	333	-	30% H <sub>2</sub> O <sub>2</sub>	Visualization	Zhao et al. (2014)
Lake Hovsgol, Mongolia	Manta trawl	333	-	30% H <sub>2</sub> O <sub>2</sub> + Fe	Visualization	Free et al. (2014)
Three urban estuary, China	Teflon pump and steel sieve	333	30 cm	Enzymatic digestion	Visualization + µ-Raman	Zhao, Zhu, and Li (2015)
Europe						
Dutch river delta and Amsterdam canals	Bulk water 2 L	-	-	-	FTIR	Leslie et al. (2017)
Rhine river, Germany	Manta net	300	-	30% H <sub>2</sub> O <sub>2</sub>	FTIR	Mani et al. (2015)
Seine and Marne rivers, France	Plankton net	80	0.1-0.35 m	-	Visualization	Dris et al. (2015)
Lake Geneva, Switzerland	Manta trawl	300	-	-	Visualization	Faure et al. (2012)
Bolsena and Chiusi lakes, Italy	Manta trawl	300	-	Hot digestion using HCL	Visualization + SEM	Fischer et al. (2016)
Danube river, Austria	Stationary conical driftnets	500	0.5 m	-	Visualization	Lechner et al. (2014)
Tamar estuary, England	Manta nets	300	-	-	FTIR	Sadri and Thompson (2014)
America						
Lake Winnipeg, Canada	Manta trawl	333	-	30% H <sub>2</sub> O <sub>2</sub> + Fe	SEM-EDS	Anderson et al. (2017)
29 great lakes tributaries, USA	Neuston net	333	20-35 cm	30% H <sub>2</sub> O <sub>2</sub> + Fe	Visualization	Baldwin, Corsi, and Mason (2016)
Goiana estuary, Brazil	Conical plankton net	300	-	-	Visualization	Lima, Costa, and Barletta (2014)
Four estuaries in the Chesapeake Bay, USA	Trawl	333	15 cm	30% H <sub>2</sub> O <sub>2</sub> + Fe	Visualization + Raman	Yonkos et al. (2014)
North shore channel, Chicago, USA	Neuston nets	333	-	30% H <sub>2</sub> O <sub>2</sub> + Fe	Visualization + SEM	McCormick et al. (2014)
Laurentian great lakes, USA	Manta trawl	333	-	-	SEM-EDS	Eriksen et al. (2013)
Los Angeles river, San Gabriel river, Coyote creek, USA	Hand net and Manta trawl	800, 500, 333	-	-	Visualization	Moore, Lattin, and Zellers (2011)

Africa						
Five urban estuaries of KwaZulu-Natal, South Africa	Conical zoo-plankton net	300	-	-	Visualization + FTIR-ATR	Naidoo, Glas-som, and Smit (2015)

Abbreviations:  $H_2O_2$ : hydrogen peroxide, HCl: Hydrochloric acid, FTIR: Fourier transform infrared, SEM: Scanning electron microscopy, SEM-EDS: Scanning electron microscope coupled with energy-dispersive X-ray spectroscopy, FTIR-ATR: Fourier transform infrared -Attenuated Total Reflectance.

However, the alkaline treatment with sodium hydroxide can damage some of the synthetic polymers as well (Cole et al. 2014). The application of potassium hydroxide (KOH) is preferable for the destruction of organic material, as it seems to attack the synthetic polymers less than the abovementioned methods (Dehaut et al. 2016). Enzymatic treatments were developed for biota-rich marine surface water samples, which allow the detection of pH-sensitive polymers (Cole et al. 2014). Single-enzyme approaches using proteinase K or mixtures of technical enzymes (lipase amylase, proteinase, chitinase, cellulase) were used for the removal of biological material, as the enzymatic digestion can be carried out under moderate experimental conditions in terms of pH and temperature. Unfortunately, the use of enzymes involves several disadvantages. Enzymatic treatments are, compared to chemical treatments, expensive and very time consuming and might not result in a complete removal of the natural debris.

### 3.4. Identification and quantification of microplastics

In most studies, microplastics are first identified visually, before an identification of the polymer type is undertaken. Larger particles can be identified with the naked eye, whereas small microplastics are identified using binocular microscopes or scanning electron microscopy (SEM). Early studies determined microplastic concentrations after visual inspection of the sample only. Depending on the efficiency of the sample treatment and particle size, the visual identification is considered to not be state of the art and often insufficient resulting in false-positive results because it is very difficult to visually differentiate the microplastics from other extracted organic and inorganic particles of similar size and shape (Löder et al. 2015). For this reason, further spectroscopic methods are needed to ensure the unambiguous identification of particles made from synthetic polymers (Wagner and Lambert (2018)).

**Spectroscopic identification** methods include Fourier transform infrared (FTIR) spectroscopy and Raman spectroscopy. These methods are based on the energy absorption by characteristic functional groups of the polymer particles. For larger particles (approximately  $>500\ \mu\text{m}$ ), FTIR can be carried out using an attenuated transverse reflection (ATR) unit as the particles need to be transferred on the crystal of the ATR unit manually (Harrison, Ojeda, and Romero-González (2012)). Coupling of FTIR instruments to microscopes such as reflectance or transmission micro-FTIR allows the detection of smaller microplastics. The use of FTIR microscopy in transmission mode is only applicable for smaller particles or thin films that do not fully absorb the Infrared (IR) beam. Moreover, special filters are required in the sample treatment that are translucent to IR radiation, such as aluminum oxide ( $\text{Al}_2\text{O}_3$ ) membranes. Both FTIR-based and Raman-based methods are limited in terms of the minimum particle size that can be determined by the physical diffraction of the light. Focal plane array (FPA)-based FTIR imaging with several detectors placed in a grid pattern, a highly promising FTIR extension, has recently been applied for microplastics analysis (Tagg et al. 2015). This technique allows for detailed and unbiased high-throughput screening of total microplastics on the whole membrane filter and enables simultaneous recording of several thousand spectra in a targeted area within a single measurement run, generating chemical images for the whole filter. Thus, the screening and analysis for the whole sample membrane filter becomes possible through combination with the FPA fields. FTIR measurements in transmittance mode are limited for particles between 10 and 20  $\mu\text{m}$ , while Raman instruments can measure particles with sizes that are one to two orders of magnitude smaller, due to the smaller wavelengths that are applied for the excitation. Identification of the polymers by FTIR and Raman is susceptible to environmentally driven changes of the polymer surface or the additive application during polymer processing. Thus, microbial fouling, soiling, adsorption of humic acids, and coloured plastics can interfere with the absorbance, reflection, or excitation of the polymer molecules and might lead to misidentification or totally prevent identification of the particles (Rocha-Santos and Duarte 2015).

The application of **pyrolysis-gas chromatography/mass spectrometry** (Pyr-GC/MS) allows the simultaneous determination of the polymer type and polymer additives by combustion of the sample and the detection of the thermal degradation products of the polymers. The identification of thermal degradation products serves as a marker that is specific for each polymer. The degradation products are separated by GC prior the detection of their specific mass to charge ratios in the mass spectrometer. In contrast to the spectroscopic techniques, Pyr-GC/MS is a destructive method, preventing any further analysis of the plastic particles. Results obtained through Pyr-GC/MS analysis are usually provided as the mass fraction or mass concentration of plastics. Therefore, the determination of particle counts is not possible due to the combustion of the sample. Thermal desorption GC/MS (TDS-GC/MS) in combination with thermogravimetric analysis (TGA) coupled with a solid-phase adsorber enables higher initial sample sizes compared to Pyr-GC/MS. For this reason, more representative results might be obtained for inhomogeneous samples with complex matrices.

SEM can be coupled with energy-dispersive X-ray spectroscopy (**SEM-EDS**), which produces high-resolution images of the particles and provides an elemental analysis of the measured objects. For SEM-EDS, the particle surface of the sample is scanned by an electron beam. The contact of the electron beam with the sample surface results in the emission of secondary electrons and element-specific X-ray radiation. Thus, an image of the particle can be created, and the elemental composition can be identified by using SEM-EDS. It is, therefore, possible to distinguish between microplastics and particles that are composed of inorganic elements, such as aluminium silicates.

Besides the methods explained above, a simple **staining method** has been reported (Shim et al. 2016). Commercially available Nile Red (NR) was applied to stain the highly hydrophobic microplastics. The NR molecule specifically binds to plastic and this molecule is only fluorescent in the presence of a hydrophobic environment. NR staining will be useful for the identification of the hidden microplastics, which could also be offered as a useful guide for the future spectroscopic analysis. The main drawbacks of the staining method are the co-staining of natural organic matters and thus pre-purification is necessary. This staining method cannot be used alone unless it is proved that the total organic matters have been fully removed by the digestion.

Alternatively, **hardness tests** are reported as inspection of the separated particles. Pressure is applied to the particles by needles or tweezers. This precludes misidentifications of microplastics with fragile carbon or carbonate particles that break during the test and are not removed or formed during the sample treatment (Eriksen et al. 2014). However, these tests are very time-consuming, do not provide exact polymer identification, and are less accurate than other instrumental methods.

## 4. Methods applied in drinking water studies

### 4.1. Brief description of applied methods

The American and Danish investigations by Kosuth et al. (2017) and CPHBusiness (2017) followed similar procedures with small differences and a brief description is provided here (Table 4).

Table 4. Comparison of American and Danish studies.

	American study (Kosuth et al. 2017)	Danish study
Sample volume	500 mL	1000 mL
Sampling bottles	- Plastic HDPE bottles	- Plastic laboratory bottles - Glass bottles with plastic blue cap
Rinsing before sampling	- Twice with tap water	- Three times with filtered water - Three times with tap water
Filtration	- Whatman cellulose filters - Filter pore size was 2.5 µm	- Brand or material of filters were not reported. - Filter pore size was 0.45 µm
Quantification and identification	- Staining with Rose-Bengal - Visual identification with stereo-microscope (8-35 times magnification) - Verification by hardness test	- No staining - Visual identification using stereo-microscope (20-40 times magnification) - Verification by hot needle test

As a part of quality assurance, blank samples were analysed in both studies. Kosuth et al. (2017) processed three types of blank samples. First, the filtrate from each sample was filtered a second time through a new filter and cleaned glassware. These blanks, referred to as filtered blanks (n=159), were carried out to make sure each sample was filtered thoroughly. Additionally, lab blanks containing only deionized water were run once each day during sample processing. These blanks were called deionized blanks (n=30) and they were carried out to account for background lab contamination from atmospheric deposition, deionized water, and glassware. Finally, bottled blanks were run by filling two empty 500 mL HDPE bottles with deionized water in the lab, just as the samples had been collected. Only the results of one type of blank sample, which was based on demineralized water filtration, were reported, and one single fibre was found in 17% of the samples. This contributed to a relatively low internal contamination. CPHBusiness (2017) processed 5 blank samples. The blanks were filtered tap water, which has undergone the same procedure as the actual samples. The background value of the 5 blank samples was determined to be 4.2 microplastic particles (range 0-6) per litre of water on average.

The German investigation, by Mintenig, Löder, and Gerdt (2014), has followed a different approach. In order to ensure a high representation of the results, the raw and drinking water was filtered through a stainless-steel cartridge filter (mesh size of 3 µm). The filters were placed in closed filter housings and could be connected directly to the tap at sampling points. Depending on the pressure and the dissolved iron content, 302 to 2572 litres of raw and drinking water were filtered.

The samples in the filter housing were then transported to the lab for further sample preparation and FTIR analysis. The filter housing was emptied using purified compressed air and refilled with filtered (0.2 µm) dilute hydrochloric acid (pH 2) to dissolve precipitated iron particles which was then easily removed by emptying the filter housing. The empty filter housings were then opened. All retained material was rinsed through a 3 µm stainless-steel filter and transferred to borosilicate glass vials where organic residues were oxidized with 20 mL of H<sub>2</sub>O<sub>2</sub> (35%, for 24 hours at 40 °C).



In the case of raw ground water, the amount of iron oxide was so high that they could not be completely dissolved by the dilute hydrochloric acid. Therefore, density separation using zinc chloride was used to separate iron oxides from potential plastics in the samples. For this purpose, the samples were rinsed from the stainless-steel filters with a 1.6 g cm<sup>-3</sup> dense zinc chloride (ZnCl<sub>2</sub>) solution directly into a separating funnel. The next day sedimented particles were drained off and the remaining material was filtered and concentrated onto aluminium filters and sent to FTIR analysis.

To avoid possible contamination, labware containing samples were covered with aluminium foil whenever possible and use of plastic tops were avoided. For all (rinse) steps in the laboratory, only MilliQ water (0.2 µm filtered) and diluted ethanol (30%), also filtered over 0.2 µm, were used. A high rate of contaminating air borne fibres was detected by testing parallel treated negative controls. These data were used to correct the amount of fibres for each sample and it was concluded that none of the fibres were originally in the ground and drinking water samples. The calculated concentrations of microplastics were very low and ranged from 0.4 to 7 microplastic particles per m<sup>3</sup> (0.0004 to 0.007 particles per litre) in ground or tap water for the ten samples.

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## 4.2. Brief description of most recently developed method

In addition, a recent study was published by Aarhus University, Institute for Bioscience which is focused on developing a reliable method for analysing microplastics in drinking water (Strand 2018). This study introduces a similar approach as reported by Mintenig, Löder, and Gerdts (2014) which includes application of sampling equipment (i.e. filters) directly to the sampling tap to eliminate the risk of contamination during the sampling. The focus has initially been on analyses of microplastic particles as fragments and fibres with sizes/lengths of more than 100µm. This lower limit of particle sizes is the same as in the previously mentioned Danish and American studies of drinking water.

### 4.2.1. Sampling method

The sampling equipment consists of a horizontal conical filter housing made of stainless steel in which there is a stainless-steel wire mesh with pore sizes of 20 µm for collecting particles. A flow meter is applied at the water outlet to measure the volume of filtered liquid. Using this system, the water samples are filtered directly from the tap at sampling point. The typical test volume was reported as 50 litres per sample. However, larger sampling volumes may also be used if considered relevant.

After sampling, the filter is back washed in two stages. First, 20 ml of filtered concentrated detergent solution (sulfo) is passed through the filter (inverse flow) which releases the microplastics from the filter surface. In the next step, the particles collected on the steel filter are then collected on a white MCE filter (mixed cellulose ester consisting of nitrocellulose, ADVANTEC) with a pore size of 5 µm by back washing the filter with 5-10 litres of filtered water. Both the rinse water and detergent solution are filtered with 5 µm filters before use.

### 4.2.2. Analysis method

The method of analysis is based on two steps. The first step consists of a visual characterization and quantification of potential micro-plastic particles on MCE filters using stereo microscopy with 10-100 times magnification. Potential microplastic particles are characterized and quantified according to their type (e.g., fibres, fragments, films, etc.), colour, and size fraction. In general, it is considered that only particles with sizes of >100 µm can be visually characterized with reasonable certainty in this manner. The second step consists of validating the visual characterization of the potential microparticle particles using µFT-IR microscopy. This allows verification of whether the particles found consist of plastic polymers or primarily of other material types (e.g. cellulose, protein, metals, etc.).

Strand (2018) used the Agilent Cary 6210/670 instrument with FPA (Focal Plane Array), which covers the wavelength range of 875-3800 cm<sup>-1</sup>. They concluded that the preferred set-up uses ZnSe filters and transmission mode. The actual identification of the composition of the particles is performed either by an experience-based visual recognition of the recorded spectra or by comparing the recorded FT-IR spectra with spectra of known materials

collected in a library database. However, the final determination of the constituents of the particles must still include a final expert assessment of the quality of the library search match. It was estimated that the current process for identifying potential micro-plastic particles with stereomicroscopy (step 1) takes about a half of a working day per sample, while  $\mu$ FT-IR analyses of 10-20 potential microplastic particles (step 2) will take about one business day.

#### 4.2.3. Contamination control

Filter housing and stainless-steel filters are cleaned before use, first by washing in detergent solution, followed by rinsing in filtered water, and finally wrapped in aluminium foil and heated to 550 °C for 2 hours in a muffle furnace to remove any organic matter. All used glassware (petri dishes, bottles, etc.) are cleaned in the same way. Black rubber stoppers used to seal the sampling system are rinsed clean before use, first in a filtered detergent solution with ultrasonic treatment and then in filtered water. During storage, sensitive surfaces and openings of the sampling equipment are packed in aluminium foil until the equipment is put into service.

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### 4.3. Assessment of the existing methods

In this section we summarize the weaknesses of the methods used in existing studies as well as possible sources of contamination which have not been considered.

- The American study; investigation performed by Kosuth et al. (2017)
  - o Only 500 mL of water sample is used. Using larger volumes of water samples is expected to reduce the significance of background values found in blank samples.
  - o There is a need to verify whether the identified microplastic particles consist of synthetic plastic polymers. It is recommended by JRC (2013) that for particles in the size range of 0.1-5 mm, a portion of the particles (e.g. 10%) in each size class, must be subjected to further analysis to confirm their identities. Such verification includes sophisticated analysis such as  $\mu$ FTIR,  $\mu$ Raman spectroscopy, or Pyr-GC/MS. Some studies (Strand et al. 2018) have shown that many fibres identified as microplastics can consist of cellulose (in paper or cotton) which cannot be referred to as microplastics.
  - o Methodological issues have previously been raised for the use of Rose-Bengal for staining naturally occurring organic particles in connection with the visual identification of synthetic fibres and fragments which are characterized as microplastics in beer samples (Lachenmeier 2015). How far the same interferences (i.e. risk of false results) can also occur when analysing tap water samples is not known.
  - o Sampling bottles made of HDPE plastic and Whatman cellulose filter papers were used in this study. It can be difficult to ensure that this sampling and filtration equipment is not contaminated with microplastics prior to use. The possibility of added contamination was not specified.
  - o As part of contamination control, three different types of blank samples at three levels of the study were used but only data for one of the three types was reported and described. Therefore, it is not possible to assess whether or not there are other types of background contamination.
- The Danish study; investigation performed by CPHBusiness (2017)
  - o As for the investigation by Kosuth et al. (2017), there is a need to verify whether the identified microplastic particles consist of synthetic plastic polymers (for more details see above). A hot needle test has been performed on identified fibres. However, there is some uncertainty about this test's reliability to distinguish between plastic and other types of material such as cellulose.

- o Sampling bottles and filtering equipment consist of plastic and filter papers. It can be difficult to ensure that this sampling and filtration equipment is not contaminated with microplastics prior to use. Possibly, the filter paper used may also be the source of fibre microplastics. The possibility of added contamination was not specified.
  - o No detailed documentation of contamination control or uncertainty analysis of data was reported. This makes it difficult to assess the extent to which background contamination related to sampling and analyses can affect the results.
- The German study; investigation performed by Mintenig, Löder, and Gerdts (2014)
  - o Sampling tubing and filter housing consists of plastic. It can be difficult to ensure that this sampling and filtration equipment is not contaminated with microplastics prior to use. The possibility of added contamination was not specified.
  - o Due to the presence of high concentrations of iron precipitates, multiple stages of sample preparation and filtering were performed before FTIR analyses. Adding multiple steps of sample processing adds to the possibility of added contamination, as well as losing particles, and may lead to under estimation of microplastics in the samples.

## 5. Project implementation

### 5.1. Participating waterworks

In total, 24 water works from different regions of Norway participated in the project. 20 of the waterworks have surface water sources and 4 water works have ground water sources. Table 5 provides the participating water works sampled and a summary information as received from them, regarding their water source type as well as their treatment process.

Table 5. Participating water works; water source and treatment process.

Code	Company or community	Water works	Raw water source	Treatment Process
A	Asker og Bærum kommune	Aurevann	surface water	Coagulation/flocculation, Dual media filtration, UV and chlorination
B	Sarpsborg kommune	Baterød	surface water	Coagulation/flocculation/sedimentation, activated carbon and sand filtration, UV
C	Trondheim kommune	BENNA	surface water	UV and chlorination
D	Øvre Eiker kommune	Eikern	surface water	Chlorination, vannglass, UV pre-filter
E	FREVAR	FREVAR	surface water	Coagulation/flocculation/sedimentation, Dual media filtration, UV (chloramine)
F	HIAS	Hamar	surface water	CO <sub>2</sub> -dosing, marmorfilter, chlorination, UV (Dose 40)
G	Asker og Bærum kommune	Holsfjorden	surface water	UV and chlorination
H	Kongsberg kommune	Hvittingfoss	ground water	Reverse osmosis filtration for removing fluorine
I	Sarpsborg kommune	Isesjø	surface water	Coagulation/flocculation/flotation, activated carbon and sand filtration, UV
J	Bergen kommune	Jordalsvatnet	surface water	Chemical precipitation using Fe, 3-media filtration (Moldeprosess), UV
K	MOVAR	MOVAR	surface water	Coagulation, DAF, Filtration (sand+filtralite), UV, Filtration (GAC), chlorination
L	HIAS	Stange	surface water	Press filter, UV, chlorination
M	Øvre Eiker kommune	Strømbo	ground water	Chlorination, vannglass
N	Bergen kommune	Svartediket	surface water	Chemical precipitation using Fe, 3-media filtration (Moldeprosess), UV
O	Trondheim kommune	VIVA	surface water	Carbonatation, UV, chlorination
P	Ålesund Kommune	Ålesund	surface water	CO <sub>2</sub> , chlorination, Marmorfilter, UV
Q	Vestfold vann	Eidsfoss	surface water	Marmorfilter, UV, chlorination + ammonia
R	Elverum kommune	Elverum	ground water	Vyredox-2 method
S	Glitre	Glitre	surface water	Siling (filtration)0,3 mm, UV, chlorination, vannglass
T	Lillehammer kommune	Korgen	ground water	Not received
U	NRV	Nedre Romerike	surface water	Coagulation/ filtration (Superpulsator), 2 Media filtrations, GAC - UV - Chlorination
V	Oslo VAV	Oset	surface water	Actic Flow, filtration, UV
W	Vestfold vann	Seierstad	surface water	Contact filtration with/Pax16, Chlorination and ammonia, (UV during mars), micronized marmor
X	VIVA IKS	Sandungen	surface water	Backwash filtration 100my, UV, chlorination, vannglass

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## 5.2. Method of sampling

### 5.2.1. Preparations prior to sampling

1 L glass Schott Duran bottles with plastic tops were used for sampling. All the bottles including tops were cleaned in a dishwasher and then rinsed three times with filtered water. The filtered water is prepared in the lab and is RO (reverse osmosis) filtered water which is filtered a second time through a 0.22 µm filter. The filtered water is stored in 10 L glass bottles and the top is covered with aluminium foil to avoid contamination.

Later the clean bottles were placed in polystyrene boxes and were sent to the water works for sampling. Labelling stickers were prepared and accompanied the bottles, which was used for labelling the bottles with name of water work, replicate number, sampling point, and date and time of sampling. In addition, a procedure explaining the step-by-step sampling procedure, as well as a reporting form accompanied the bottles (see appendix 1).

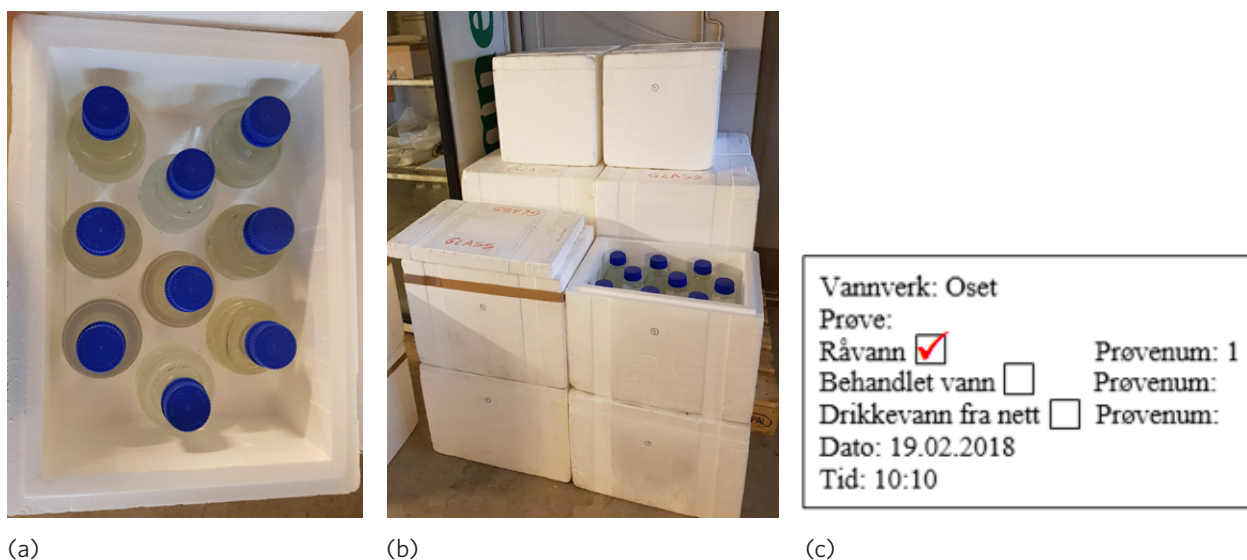


Figure 1. Illustration of equipment used for sampling; (a) and (b) glass bottles used for sampling and polystyrene boxes used for packaging and delivery; (c) sample label prepared by NIVA for labelling the bottles prior to sampling.

### 5.2.2. Water sampling procedure

Three sampling points were chosen from each water work which includes the entrance to the water treatment plant (i.e. raw water), at the end of treatment process (i.e. finished treated water), and from the distribution system (i.e. drinking water). Information regarding treatment processes as well as sampling points for each water work are summarized in Section 6.1.

The samples were taken in three parallels and in total 9 samples were taken from each of the water works. The water works were asked to find sampling points with minimum dust and air movements to avoid air borne contamination. The responsible person for sampling was advised to wear non-synthetic clothing during the sampling. To ensure that no contamination from the sampling tap is included in the sample, the sampling tap was opened to the maximum, stayed open for 1 minute, and then closed. This was repeated three times. Then the tap was opened and stayed open for 1 minute. The bottle top was opened, and the bottle was rinsed with sampling water two times before it was filled for the third time and the top was immediately closed. The responsible person was asked to hold the bottle top upside down during the sampling and to not leave it on any surface.





Figure 2. Pictures were taken by water works during sampling; (a) sampling from treated water at the plant; (b) and (c) sampling from distribution network, different sampling points were used by different water works. (with kind permission from Annie Bjørklund, Frank Herland (a) and Mads Mellingen (c) from Bergen Vann KF and Kaj-Werner Grimen (b) from MOVAR)

### 5.2.3. Re-labelling of samples prior to analysis

After reception of the samples at NIVA's lab each bottle was assigned a random number. After registration of the origin of the bottle and the random number, the labels were removed prior to delivery to the person doing the analysis. This way, the samples were completely anonymous to the analysing person and were analysed randomly. Figure 3 shows the process of random coding.

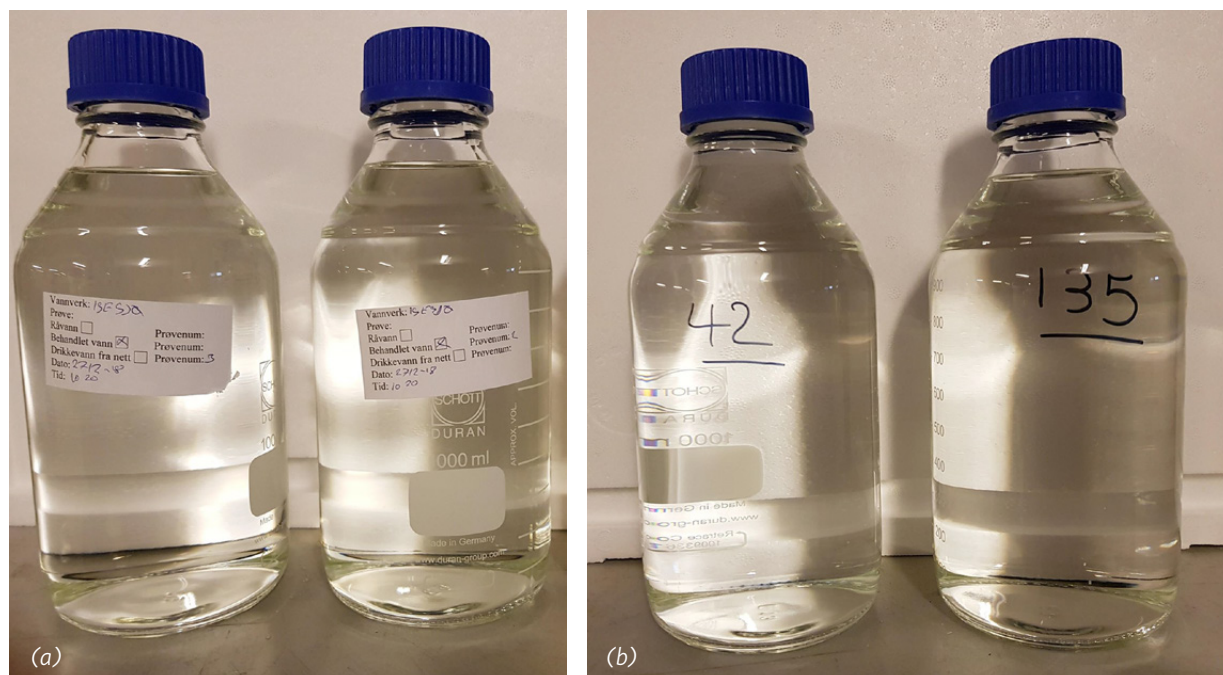


Figure 3. Re-labelling of samples before analysis; (a) Samples as received and (b) a random number attributed to the registered samples.

### 5.2.4. Issues reported during sampling

Throughout the project implementation, it was noticed that some of the particles from the polystyrene foam of the packaging boxes used for transport were sticking to the bottles, the bottle top, and most probably to the cloths and hands of the person responsible in the sampling process. This was also pointed out by one of the participating waterworks. Therefore, this was considered as a possible source of procedural contamination during data analysis.



Figure 4. Polystyrene particles from packaging were sticking to the sampling bottles (with kind permission from Ryan Mathisen from Vestfold Vann IKS).



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## 5.3. Method of analysis

The following procedure was followed for sample analysis:

- **Filtration:** Water bottles were selected at random and filtered under vacuum onto Whatman glass microfibre filter papers without binders, grade GF/C, 47mm diameter (Provider VWR, art No. 513-5227) using a Buchner set up. Prior to filtering, each filter paper was examined under a microscope for suspected contamination and placed in a closed petri dish until required. For each bottle the water was decanted slowly into the receiving funnel. Once the bottle was empty it was flushed three times with pre-filtered water into the funnel. The funnel was then rinsed with pre-filtered water to make sure no particles had adhered to the apparatus. Finally, the filter paper was returned to the petri dish and the petri dish closed with the lid until visual analysis was carried out. The filters were stored in sealed petri dishes at room temperature prior to analysis.
- **Visual analysis:** Samples were analysed using a stereomicroscope with Infinity 1-3C camera and INFINITY ANALYZE and CAPTURE software to take pictures and to measure size (longest and shortest) of all particles found. All filter papers were observed, and any particles identified and  $> 60 \mu\text{m}$  in diameter were marked on the filter paper. The lower size limit of  $60 \mu\text{m}$  is due to the fact that it was found to be impossible to properly distinguish plastic and non-plastic particles of smaller diameter. Once all filter papers were marked, photos and measurements were taken and saved, with reference to the sample ID. Particles that resembled contamination from the methodology (as e. g. typical polystyrene fragments) were excluded, as well as fibres clearly identified as cotton. Later, the findings will be validated using micro-FTIR.
- **Blanks:** Blanks were carried out to test for methodological contamination. At the start of each filtering period three blanks were analysed, and after 20 samples another 3 blanks were analysed. When there was an extended pause in the filtering three blanks were conducted again.
- **Contamination control:** Strict contamination controls were carried out during processing and analysis. Steps taken to avoid contamination included: use of pre-brushed cotton laboratory-coats, clean laboratory conditions in an enclosed room, filtered ( $0.22 \mu\text{m}$ ) RO-water, and washing of all glassware including rinsing with filtered RO-water. Also, if filter papers were exposed to the laboratory atmosphere during microscope work, an additional filter paper was left exposed for the same duration.
- **Accounting for bias:** All bottles were labelled blind before being received by the laboratory. Within the laboratory, samples were randomized when filtering to increase unknowns. To maintain an order of impartiality, the petri dishes were labelled on the bottom. When visual analysis was carried out, bias was removed, and all filter papers treated the same (including blanks).

## 6. Results and discussion

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### 6.1. Accuracy and precision of the method

#### 6.1.1. Accuracy

Accuracy describes the ability of an analytical method to find the true concentration. Or, when taking into account random deviations, that the mean of the normal distributed findings is the true concentration. Thus, standards with known concentrations must be analysed and the results of the analysis be compared with the true concentration. However, for microplastic particles in water, no standards are available. Therefore, blank samples were prepared by filtering deionized, ultrapure water through 0.22 µm filters and collecting them in bottles that had undergone the same cleaning procedures as the bottles for sampling. The blanks were analysed in between the regular samples such that, usually, three sample bottles were analysed between two blank bottles.

For the blanks, it is, at first, expected that no microplastic particles should be detected at all. However, although special care had been taken to avoid contamination of the samples during analysis from the air, microplastic particles were found at very low concentrations. The hypothesis that this was due to contamination from air was supported by the fact that cotton fibres were found, at the same very low concentrations as microplastic particles.

In total, 72 bottles blanks and 216 bottles samples had been analysed during the project. The results from the blanks were analysed statistically, comparing the results obtained during the different sampling campaigns. No differences were found for the two campaigns. Thus, it is legitimate to determine the average from all blanks and the random deviations, expressed in the standard deviation and the confidence interval. This revealed an average blank concentration of 0.5 microplastic particles per litre in the blanks, with a standard deviation of 0.82/L (n = 72).

While in analytical chemistry usually 95% or 99% confidence intervals are used, we decided here to use 33% confidence intervals for data interpretation. This is quite commonly used in environmental sciences and due to very low concentrations as well as many factors impacting variability of environmental samples.

For the blanks, the standard deviation and the number of 72 blanks analysed results in a 67% confidence interval of 0.10/L which is termed Limit of Blanks for 67% confidence, LoB(67%). In other words, when measuring a concentration in a blank sample (as this is not prone to errors during sampling) of 0.1/L and saying “there is something in the blank sample” the probability to be wrong is still 33% and the probability that there is not something in the samples is 67%.

It is questionable whether those 0.5 microplastic particles per L result from contamination during the preparation of the blanks (i.e. whether the blanks were not really particle free or the bottles were not absolutely free from particles). Or, alternatively, whether the microplastic particles found in the blanks resulted from contamination during the process of analysis in the laboratory.

Considering all the steps of production of the blanks and of the analysis, it is concluded that the microplastic particles in blanks most likely result from contamination during analysis. Thus, as the blanks were analysed regularly between the real samples, it must be concluded that the real samples received the same contamination, on average, during analysis. It is therefore straightforward that the average concentration found in the blank samples (and their standard distribution) is subtracted from the findings in the real samples. This had been done accordingly, and the standard deviation for blank samples was taken into account using Gauss' error propagation when confidence intervals for the real samples were calculated (see the following sub-chapter).

#### 6.1.2. Precision

For determination of the method precision, for all sampling points triplicate samples, i.e. three bottles each, were taken and analysed in the lab as described previously. From that, a method standard deviation was obtained. This way, errors were covered and determined for the whole procedure from sampling at the water works and in the distribution system, respectively, via the filtration process in the lab and the microscopic analysis. The results are given in Table 6. It can be seen, that the method standard deviation and the 67% confidence interval decreases slightly from raw water to finished water to drinking water. The average 67% confidence interval is 0.81

The confidence interval of 67% describes the interval below and above the measured concentration, in between this the probability to fail when expecting the true concentration in that interval is 33% or less. This is referred to as “likely”.

In the following, a conservative estimation of the risk to fail when giving the confidence interval was done, as is common in such investigations. As all samples were taken in triplicate (three bottles) and the three samples were analysed separately, a standard deviation for the triplicates was calculated. That standard deviation was compared with the method standard deviation for the respective group (i.e. raw water, finished water, and drinking water). The larger of the two standard deviations was then used to calculate the confidence interval for the mean from the three triplicates.

Finally, as the average from the three blanks was subtracted from the average of three triplicates, a combined confidence interval was calculated, which takes into account Gauss’ error propagation. Thus, concentrations and confidence intervals given in the following chapter reflect the combined uncertainty resulting from the sampling, the analysis of the blanks, and the analysis of samples in triplicate.

When the confidence intervals overlap with the zero line, it must be concluded that no microplastic particles were detected in the (triplicate) sample, on a 67% confidence level. When the average of the triplicate samples (i.e. the data point in the diagrams) is below the limit of quantitation LoQ (67%) of 4.8/L, then the concentration of microplastic particles in the samples are so low that quantification cannot be made.

The limit of detection for 67% confidence, LoD(67%) is obtained as the sum of the 67% confidence intervals of the blanks and the average of the respective confidence intervals of the samples and thus obtained as 0.91/L. In other words, if in triplicate samples an average concentration of 0.91/L microplastic particles is found, then the probability to fail when saying that microplastic particles were found is still 33% and the probability that there are microplastic particles in the samples is 67%.

The limit of quantitation generally is defined as the lowest concentration “which can be quantitatively determined with precision and accuracy appropriate to analyte and matrix considered” (AOAC, 2002). In analytical chemistry, most often ten times the standard deviation is used. However, the factor ten is historical and is not based on statistics and often factors of five or six are used as well. Here we use a slightly different approach, requiring the probability that microplastic concentration in a sample can be distinguished from blanks is 99.99%. Using that approach, the LoD is found as 4.1/L.

Table 6. Method precision for three groups of samples

Group of samples	Method Standard deviation [1/L]	Number of triplicates	Confidence Interval 67% [1/L]
Raw water	1.51	24	0.86
Finished water	1.42	24	0.81
Drinking water from distribution	1.34	24	0.76
Limit of Blanks LoB(67%)	0.10 / L		
Limit of Detection, LoD(67%)	0.91 / L		
Limit of Quantitation, LoQ	4.1 / L		



## 6.2. Microplastic particles in raw water

Figure 5 gives the measured concentrations of microplastic particles in the raw water of the participating waterworks. For simplicity, the overlapping of the confidence interval with the zero line means that no microplastic particles were detected (the error to fail when concluding detection is higher than 33%). This is the case for 14 out of 24 waterworks. However, for all samples, the average of the triplicate bottles analysed was below the limit of quantitation of 4.1 microplastic particles/L.

From these results, it must be concluded that microplastic particles were most likely not present in the raw water samples analysed. If any were present in 10 out of 24 samples, they were not quantifiable.

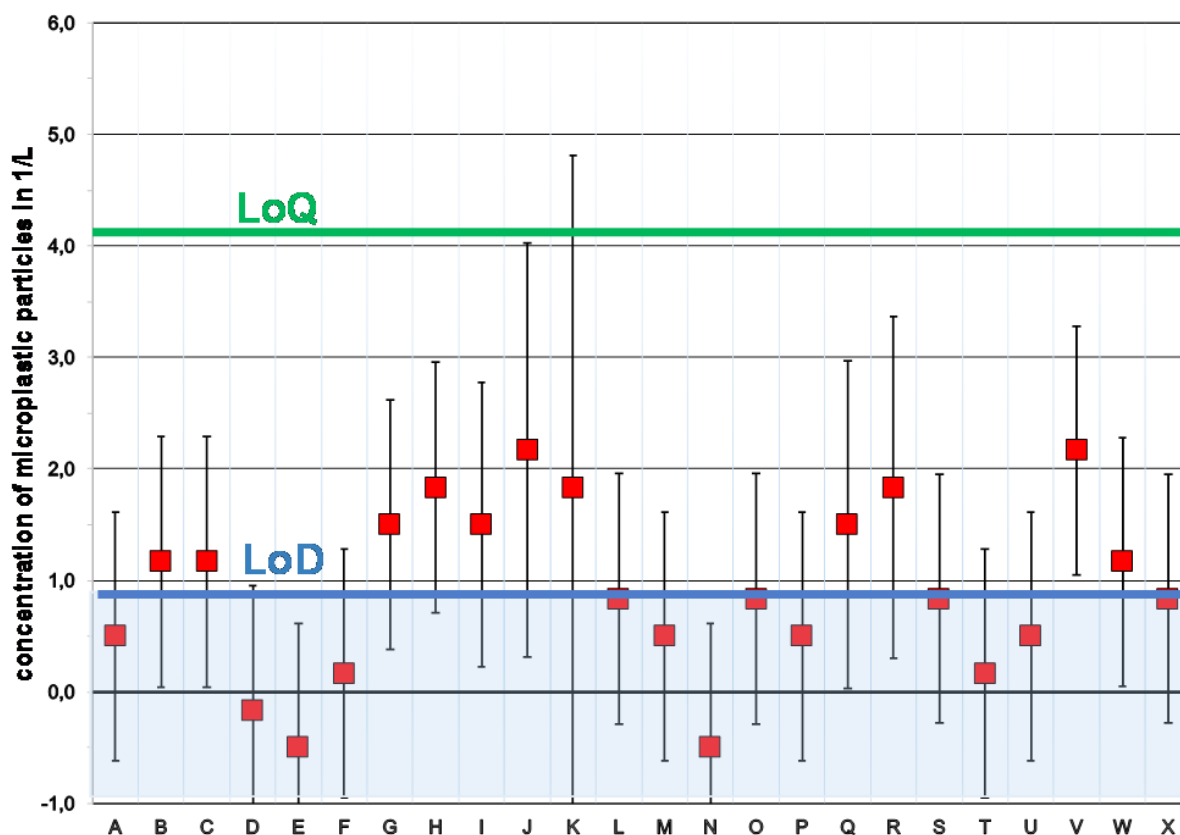


Figure 5: Measured microplastic concentrations in the raw water of the waterworks A-X, and 67% confidence intervals. The names of the waterworks are given above in Table 5.

## 6.3. Microplastic particles in treated water

Figure 6 shows the concentrations of microplastic particles found in triplicate samples of treated water from the participating waterworks. In 20 out of 24 triplicate samples, the 67% confidence bands were overlapping the zero line. Furthermore, for the four triplicate samples remaining, the data points are above the LoD(67%) and far below the limit of quantitation, LoQ.

Thus, it must be concluded that microplastic particles were most likely not present in the finished treated water samples analysed. As for the raw water, if any were present, they were not quantifiable.

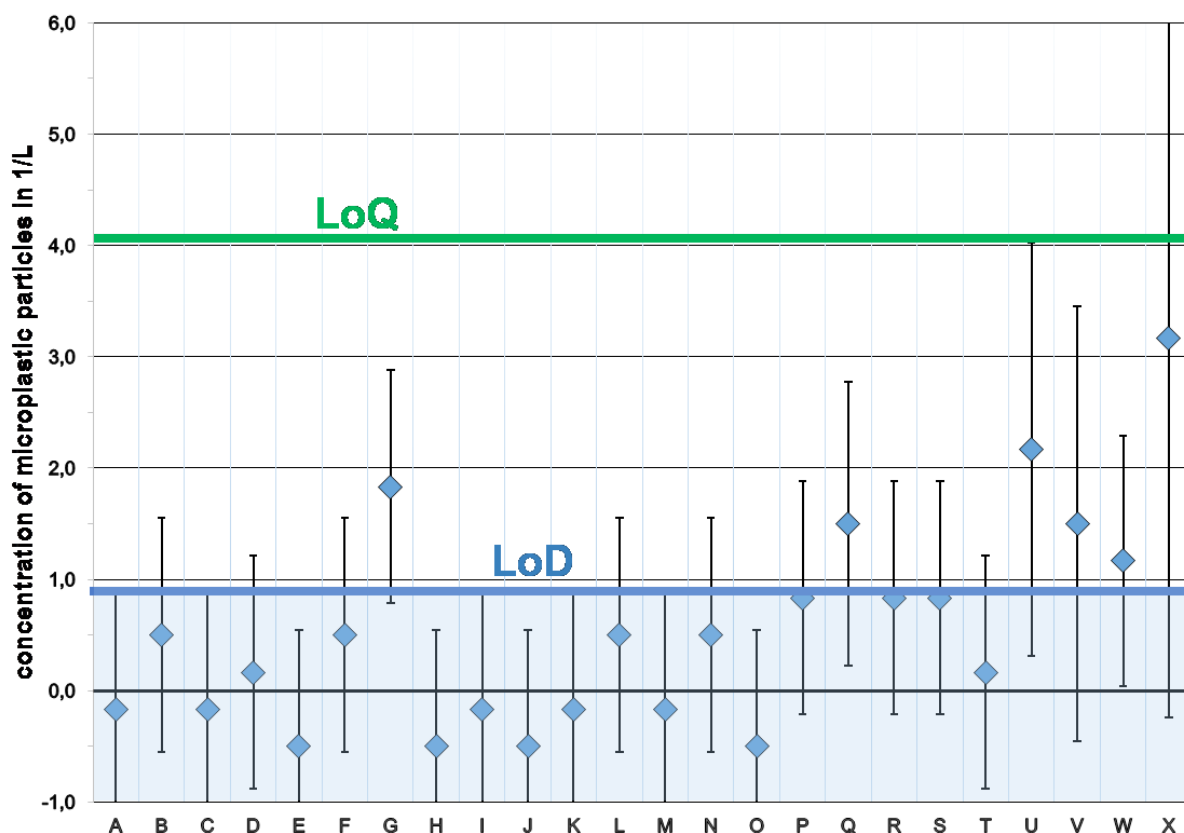


Figure 6: Measured microplastic concentrations in the finished treated water of the waterworks A-X, and 67% confidence intervals. The names of the waterworks are given above in Table 5.

## 6.4. Microplastics in drinking water from distribution system

Figure 7 gives the concentrations of microplastic particles in samples of drinking water from the distribution system of the participating waterworks. For 16 out of 24 triplicates, confidence intervals were overlapping the zero line, i.e. no microplastic particles were detected in those samples.

Apart from one sample (waterworks N), all averages from the triplicates were below the limit of quantitation of 4.1/L. A closer look at the circumstances at which these respective triplicates had been taken revealed that in this case the conditions for sampling in the distribution system had been far from ideal. In fact, the sampling point was a place where a contamination from air was likely to occur.

Conclusively, also for the finished drinking water, it must be concluded that no microplastic particles could be detected. If any had been present, they were present at such low concentrations that they could not be quantified. For the one location where the microplastic particle concentration was above the limit of quantitation, this was attributed to contamination during sampling.

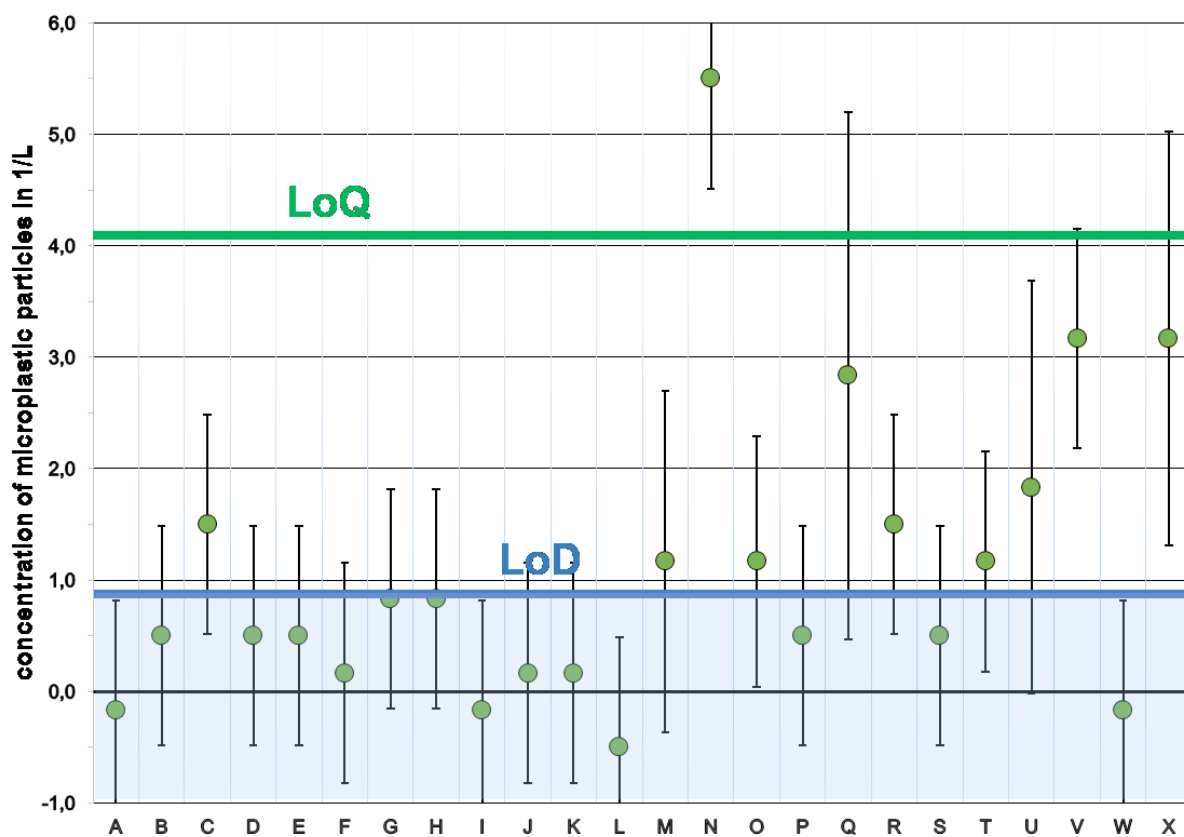


Figure 7: Measured microplastic concentrations in the drinking water from the distribution system of the waterworks A-X, and 67% confidence intervals. The names of the waterworks are given above in Table 5.

## 7. Evaluation of human health risks

### **We are exposed to plastic particles**

Humans are exposed to plastic particles such as nano- and microplastics through foods and air. Currently, no analytical methodology for measuring nanoplastics exists. However, microplastics have been found in fish and seafood, beer, honey, and bottled water. Fish and seafood contain the highest amounts of microplastics. However, most of the microplastics are located in the gastrointestinal tract and therefore cleaned seafood and fish, where the gastrointestinal tract is removed, will only contain small amounts of microplastics. Whereas shellfish, such as mussels and oysters, where the gastrointestinal tract is not removed can be a source of larger amounts of microplastics.

### **Plastic waste and plastic particles are a threat to the environment**

In recent times, there has been a focus on harmful effects of plastic waste, including nano- and microplastic particles. It has been shown that microplastics are harmful to wildlife, both below and above the sea surface, and that they can be transferred along the food chain. However, there has been less research on the potential hazardous effects of nano- and microplastics in humans.

### **Human health effects are unknown**

In 2016, the European Food Safety Authority (EFSA) published a report on microplastics and nanoplastics in food with a particular focus on seafood. The EFSA concluded that there was insufficient data on the occurrence, toxicity, and uptake to conduct a full risk assessment. Currently, it is therefore not possible to conclude whether exposure to nanoplastics and microplastics are hazardous to humans. The Norwegian Scientific committee for Food and Environment is currently working on a summary of the status of knowledge of the occurrence of microplastics and potential health implications, which will be published in 2019.

### **The majority of the microplastics are not absorbed in the body**

Particle size is likely to be the most important factor in determining the extent and pathway for uptake, although, composition, surface charge, and hydrophilicity are also thought to affect the uptake. No in vivo human data on the uptake of microplastics are available. However, existing literature in mammals indicates that microplastics of  $>150\text{ }\mu\text{m}$  are not absorbed, therefore, only local effects on the immune system and inflammation are expected for these particles. For particles  $<150\text{ }\mu\text{m}$  it is likely that only a fraction is absorbed in the intestine, causing systemic exposure.

### **Microplastics can contain contaminants and pathogenic bacteria**

Microplastics can contain additives, such as bisphenol A and phthalates. It has also been shown that microplastics can contain relatively high amounts of contaminants such as polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs). Calculations have shown that even a large portion of mussels, which are eaten without removal of the digestive tract, will only have a small effect on the exposure to additives and contaminants. In addition, it has been shown that pathogenic bacteria can colonise the microplastics. However, the consequences to human health are unknown.

### **The drinking water in Norway is safe**

The current survey shows that no microplastic particles or at concentrations far below the limit of quantification were detected in Norwegian drinking waters, both before and after water treatment or in water from the distribution system. Due to methodological limitations, only microplastic particles of  $60\text{ }\mu\text{m}$  and above have been measured. There is a need for development of standardized analytical methods to detect and identify plastic particles of  $100\text{ }\mu\text{m}$  and smaller to verify that the drinking water also only contains low levels of these smaller plastic particles.

At present, there is little evidence of the negative health effects in humans due to exposure to plastic particles, although further research is necessary to rule out that these particles are not hazardous to humans. Considering the low amounts of microplastics measured, the consumption of tap water will only to a small extent contribute to the total exposure of microplastics. Therefore, there is no need for concern for consumption of tap water in regard to exposure to microplastics and human health effects. Nevertheless, since plastic waste and plastic particles have proven to be a major environmental threat, it will be important to reduce the release of plastic in the future.

## 8. Conclusions

In the course of the project, 72 triplicate samples from 24 waterworks in Norway and 72 blanks were analysed for microplastic particles. This enabled the authors to define the limitations of such an analysis and to conclude on the limits of detection and limits of quantitation. These limits are dependent on the sampling and the analytical procedure and are considered to be valid when special care has been taken to avoid contamination during sampling, triplicates of 1 L were sampled and analysed, and when analysis has been done as in this study.

In the majority of all samples from raw water, treated water, and drinking water from the distribution system analyzed, microplastic particles could not be detected. In very few samples where the concentration was found above the limit of detection of 0.9/L, concentrations were far below the limit of quantitation (67%) of 4.1 microplastic particles per litre, except for one case. In that one case it was very likely that contamination from air had happened due to unfavourable sampling conditions.

From the findings in this study, it is concluded that concentrations of less than 4.1 microplastic particles per litre from triplicates should not be given or used for comparison. Whenever analysis is done to elucidate a possible contamination of water, special care must be taken in the setup and in the conductance of the analysis. Furthermore, the limits of detection and of quantitation must be taken into account in the design of the experiment, the sampling, and in the decision about the number and volume of samples to be analysed.

In the current study, no microplastic particles could be detected or quantified in the drinking water of the water works who participated in the study (at a 67% confidence level). The participating water works had been selected since their drinking water sources were anticipated to have the highest probability for all Norwegian water works to be polluted with microplastic particles. Therefore, it is very likely that the finding that no microplastics could be detected in the drinking water will apply to all drinking water in Norway. There is the possibility that the drinking water in Norway could contain microplastic particles at extremely low concentrations that are below the detection limit, however, these low concentrations do not provide a health risk.

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## 10. Appendixes

### 10.1. Appendix 1: Sampling procedure and reporting form



NOTAT

17. april 2018

Utarbeidet av NIVA v/: Amy Lusher, Mona E. Dadkhah, Wolfgang Uhl

**Sak:** Prøvetaking av drikkevann for MIKROPLAST-analyse

**Mål:**

Å kartlegge konsentrasjon av mikroplast i drikkevann ved prøvetaking og analyse av **råvann**, **behandlet vann** og **drikkevann (fra nett)** fra et utvalg vannverk.

**Prøvetaking:**

Utstyr:

- 9 x glassflasker (Schott Duran) med plast topp (1000 ml volume). Flaskene leveres av NIVA. Flaskene ble vasket og spesielt beh for prøvetaking av drikkevann og for analyse på mikroplast.
- Rapporteringsark som skal sendes tilbake til NIVA sammen med prøvene.
- Stoppeklokke



Kvalitetssikring ved prøvetaking:

- Vennligst bruk klær lagret av naturlige fiber når du tar prøver (for eksempel 100% bomull). **IKKE BRUK SYNTETISKE FLEECER.**
- Velg et prøvetakingssted som er minst mulig utsatt til både støv og luftbevegelser.

Metode:

1. Merk hver flaske med klistremerker (forberedt og levert av NIVA). Skriv på dato, tid, prøvenummer og vannverkets navn. **Husk å kryss av for enten råvann, behandlet vann eller drikkevann** (se eksempel).

Vannverk: Oset	
Prøve:	
Råvann <input checked="" type="checkbox"/>	Prøvenum: 1
Behandlet vann <input type="checkbox"/>	Prøvenum:
Drikkevann fra nett <input type="checkbox"/>	Prøvenum:
Dato: 19.02.2018	
Tid: 10:10	

2. Hold flasken lukket først.
3. Åpne kranen på prøvepunktet maksimalt og la vannet renne i et minutt.
4. Lukk kranen.
5. Gjenta (3) og (4) tre ganger.
6. Åpne kranen og la vannet renne i et minutt.  
(I fall det er i et hus, vent så langt at vannets temperatur er konstant.)
7. Åpne en flaske forsiktig og hold lokket nedvendt hele tiden. Husk at lokket skal **aldri** legges av på en overflate.
8. Fyll flasken til omtrent 50%, skyll og kaste innholdet. Gjenta dette to ganger.
9. Fyll opp flasken og skru på lokket forsiktig.
10. Fra hvert prøvepunkt skal prøvene tas i triplikater.  
Gjenta den andre og tredje flaske.
11. Fyll ut rapporteringsskjema.
12. Send prøvene sammen med utfylt rapporteringsskjema tilbake til NIVA.  
Vennligst bruk boksene som flaskene ble sendt til dere.  
Klistremerker med returadresse er vedlagt.

## Rapporteringsskjema:



Innhenting av vannprøver: mikroplast i drikkevannet i Norge

Dato: \_\_\_\_\_

Tid: \_\_\_\_\_

Lokasjon: \_\_\_\_\_

Navn: \_\_\_\_\_

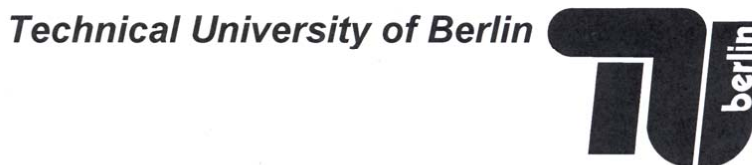
Eventuelt: \_\_\_\_\_

Send to: Norwegian Institute for Water Research (NIVA), Gaustadalléen 21, NO-0349 OSLO

Attention to: Mona Eftekhar Dadkhah (MOD)

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## 10.2. Appendix 2: Evaluation and quality assurance check



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Date: 30.08.2018

Norsk Vann Report 211/2015 of NIVA (by W. Uhl and others) on microplastic (MP) in raw and drinking waters of Norway, August 2018.

Here: Evaluation and quality assurance check.

Dear all,

the report was received in a final version on August 27<sup>th</sup>, 2018 and was carefully evaluated in view of the general approach and the efforts in quality assurance in sampling and analyzing water samples for microplastic particles out of a number of samples from raw waters, drinking waters and of samples from the distribution network.

I want to mention, that I am presently the coordinator of a very large German project on microplastic in freshwater systems (MiWa), including sampling, sample treatment and identification of the MP. This project, running for about 2 years, is directed to develop reliable analytical tools for MP and includes 12 reputed German research groups on the this topic, with one group working on MP in water supply systems.

The first parts of the report are comprehensive summaries of the state of science and the methods used to sample microplastic particles (defined with a diameter below 5 mm) from waters, as well as sample treatment and the final analytical methods. This review shows the high complexity of analyzing microplastic particles and the lack of harmonized or even standardized methods. The overall procedures for microplastic sampling, sample treatment and analytical determination (either mass and identity of MP, their size distributions incl. the identity) are still in development and it will take a few years until until standardized methods are available and we have confidence in the data produced.

Within the last few years a number of publications on the presence of MP in drinking waters (from public supplies and in bottled water) raised wide-spread concerns about the pollution of and the consumption of these waters. The methods used were not validated at all and it can be assumed, that most data were false-positive, due to the lack of careful procedures and the contamination of samples by MP particles in all stages of the analytical methods. For example, blank values are not reported, but MP are ubiquitous in our life as well as in the laboratories and contamination is a critical issue in MP analysis.

The report presented on the findings of MP in Norway is based on a very well documented approach in sampling and sample analysis. To my knowledge, the different steps including the microscopic identification was taken care of the pitfalls mentioned above and the concentrations obtained are reliable. The statistical treatment is correct, indication a limit of quantification (LoQ) of 4.1 particles per L for particles above a diameter of 60  $\mu\text{m}$ . This limit represents the threshold of concentrations that may be reported, if care is taken as in the study presented and when 3 L of sample are analyzed.

The findings of real waters clearly show that MP-particles cannot be detected in the majority of finished drinking water in Norway or are at least below the limit of quantification (except of one out-lyer).

It has to be stated, that particle removal is the first and most important step in drinking water treatment, as we want to remove all particles at once and the microbiological contamination is a part of the total particle content. Our removal processes in drinking water treatment are taking care of the near-to-complete turbidity or particle removal, with defined threshold values for the finished water (including the bacterial pathogens).

In summary, this report provides a validated approach for a reliable screening of MP in water samples from raw and finished waters in Norway.

With best regards



Prof. em. Dr. Martin Jekel











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